COAL CHARACTERIZATION RESEARCH: SAMPLE SELECTION, PREPARATION, AND ANALYSES

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INTRODUCTION

There are few procedures available to predict process responses or to assess the relative values of coals from fundamental coal properties. Therefore, a comprehensive coal characterization program has been established at Exxon Research and Engineering Company, Baytown, Texas to evaluate coals as process feedstocks. The objective of this program is to relate fundamental coal properties and process response patterns by analyzing and testing a large suite of U. S. coal samples.

This report defines the technical rationale behind the coal characterization program and describes the procedures used in the selection, preparation, and testing of the coal library samples.

RATIONAL APPROACH TO COAL CHARACTERIZATION

The principle goal of a coal characterization research program should be to develop procedures that define the minimal testing required to evaluate coals as process feedstocks. The alternatives available are (1) to test any 'unknown' coal (coal X) empirically in a commercial or small-scale process or (2) to develop information about the fundamental relationships between coal properties and process responses, and to use this information and appropriate analyses of coal X to predict its response.

The empirical technique is direct and provides an unequivocable answer about the response of the tested sample. The results, however, cannot be extrapolated to other samples with different characteristics. This is particularly critical during exploration programs where the coal characteristics may change significantly over the areas being evaluated. Though development of fundamental understanding is more difficult, it provides much greater flexibility in subsequent testing. Only a program designed to relate fundamental properties of a broad range of coals to their process responses has value to the coal community.

Criteria Used in the Selection of a Set of Coal Samples for Research

Coal characterization efforts aimed at predicting coal responses from fundamental properties must follow a rational scientific approach (viz. characterization by rank, type and grade), if they are to yield extrapolatable results[1]. Major differences between "typical" coals in the U.S. are rank-related. Therefore, we employed criteria for selecting our coal samples that maximized coal rank variability. Variations in type, though purposely minimized could not, however, be eliminated and are dealt with as part of this study.

A set of research samples was selected according to the following criteria:

(1) Broad range in rank. In effect, this maximizes variation in organic chemical composition, principally the elements C, H, and O. High rank coals (anthracites) were excluded, because they are of little economic value from a synthetic fuels standpoint.

(2) Vitrinite-rich. Vitrinite is the predominant maceral in most U.S. coals and for that reason its reactivity is of most concern in utilization. Also, the properties of vitrinite vary progressively with rank.

(3) Low in inorganic matter content (preferably less than 10%). This restriction minimizes problems which can arise during analyses and subsequent calculations of the properties of the organic components.

(4) Geographically and geologically diverse (within the continental U.S.). Samples were obtained from the major coal-bearing regions in the United States so as to include different metamorphic histories and geological ages.

(5) Fresh and unweathered.

The optimum range in rank variation of vitrinites is best defined by the classical H/C vs O/C diagram from van Krevelen[2]. When published data (from the Pennsylvania State University coal-data base [3] for high vitrinite (>80%), low mineral-matter coals (<10%) from various regions in the U.S. are plotted on a van Krevelen diagram, a broad band is obtained (shown in Figure 1). From along the length and breadth of this band a random set of coals was identified and then located for collection. Sixty-four samples were ultimately selected for the research study. Two foreign samples were also added to the library. Oistribution of these 66 samples on the van Krevelen plot is shown in Figure 1.

The coal samples were collected by experienced coal geologists directly from freshly exposed seam faces. Where possible, the samples were selected from lithotypes rich in vitrain. Some samples were obtained as run-of-mine samples, providing they were recently mined and free of extraneous rock.

Most samples were collected as coarse lumps (larger than 5-6 cm). Samples were sealed within heavy gauge, polyethylene bags, placed in epoxy-lined containers (when possible, they were placed under water) and shipped. Any information that might ultimately be pertinent to sample quality was logged. Upon receipt, the samples were stored in a cold room (at 30-40°F) until preparation.

SAMPLE PREPARATION

Initial Bulk Sample Preparation

The procedure for the general workup and subsequent characterization of coal samples for this study is summarized by Figure 2. To minimize oxidation during preparation, samples were handled in nitrogen-filled glove boxes (where possible). Maintaining water filled pores minimized exposure of the internal surface to air and prevented irreversible pore collaspe. The initial sample was inspected and a hand specimen was taken for display. Extremely coarse (>5 cm), particles were broken by hammer to about a 5-cm top size. The coal was then washed on a 16 mesh screen (or 100 mesh if the sample was fine) to remove any extraneous mineral matter, debris and fines. Such physical beneficiation to provide sample consistency can be performed when seam-representive samples are not required, as in studies such as this. Subsequently, the coal was surface dried under nitrogen, frozen for at least 4 hours in solid CO_2 ("dry ice"), and then crushed to -4 mesh

using a swing-hammer mill. Freezing the samples minimizes the risk of thermal change, oxidation, and volatile release due to heat generation during size reduction.

The coal was then riffled into 3-kg representative splits. Bottles containing these splits (and all subsequent samples) were placed in a bell jar with the bottle lids loosely in place. The system was evacuated and refilled with nitrogen three times to replace any air. All bottles were capped with tightly fitted lids which were then secured with tape. Sample identification numbers are permanently etched into the bottles. Samples were stored in the manner indicated in Figure 2.

Preparation of a 16 x 100 Mesh Analytical Sample

One of the 3-kg samples of the -4 mesh coal (the "working sample") was stage-crushed using a mechanical gate mill (a "coffee grinder") in a nitrogen filled glove box. After passing through the coffee grinder, the coal was screened on a 16 mesh screen and the top size recycled. The process was repeated until all the sample passed through the 16 mesh screen. This stage crushing maximized the particle size consist of the crushed coal.

The -16 mesh material was wet-screened on a 100 mesh screen by washing repeatedly with water. The fines were discarded. Approximately 70 to 80% of the 3 kg split was retained by this method. Wet screening physically beneficiates the coal by removing fines. Excessive fines are undesirable in certain analytical tests (i.e. HGI, petrography and some process response determinations). Also, removal of fines decreases fusinite and mineral content of the sample, since these components preferentially report to the fines fraction during size reduction.

Excess water left on the 16x100 mesh coal from the washing procedure was removed by placing the wet coal in a 25 cm Buchner vacuum filtration assembly inside of a nitrogen-filled glove box. The aspiration forces nitrogen through the sample and dries the surface of the coal, so that it can be riffled, without exposing the pore structure to air (oxygen).

Four splits containing about 500g of 16 x 100 mesh coal were obtained using a mechanical riffling device. Two splits were stored for future work. The other two fractions were equilibrated in nitrogen at 50% relative humidity and used for all of the analyses. Equilibration at 50% relative humidity is required to obtain reproducible weighings (50% relative humidity is typical for most labs). Nitrogen is used as the equilibrating gas to minimize oxidation. The 50% relative humidity nitrogen was obtained by bubbling nitrogen through water under 2 atmospheres of pressure and subsequently expanding the saturated nitrogen through a regulator to one atmosphere of pressure. The partially moist nitrogen was passed through a manifold into a number of bottles containing coal, and after passing through the coal, was vented. A top loading balance was used to record the weight of each of the bottles of coal as a function of time. When the weight stabilized, equilibration was achieved.

Some coal analyses require coarse-sized coal and other analyses require pulverized (-60 mesh) coals. Splitting the 16 x 100 mesh fraction into a 16 x 60 mesh fraction for coarse analyses and -60 mesh fraction for chemical analysis would not have been valid since coal components segregate to different size fractions. Therefore an aliquot of the 16 x 100 mesh coal was pulverized to -60 mesh.

Preparation of Aliquots for Analysis

Numerous aliquots of about 5g each were riffled in a nitrogen-filled glove box using a rotary riffling device. The rotary system is far superior to other sample splitting methods in that it affords better reproducibility between splits and allows a rapid production of multiple aliquots. Each aliquot was used for only a few tests or analyses. By preparing many aliquots at the outset, oxidation, contamination, or non-representativeness often associated with repeated handling of bulk analytical samples, is avoided.

ANALYTICAL PROCEDURES AND DATA QUALITY

Analyses and tests performed on the samples are shown in Figure 2. Most were performed according to ASTM standardized procedures. Brief descriptions of non-standard tests are described below.

1. Extraction with citric acid and benzene/ethanol azeotrope.
2. Assessment of acidic functionality by $Ca(OH)_2$ and $Ba(OH)_2$ ion exchange.
3. Density by helium pycnometer and by water displacement.
4. Gasification in a small fluidized bed.
5. Pyrolysis in a rapid-heating, fixed bed system. Yields of char, tars, water and gases assayed. Product properties assessed.
6. Liquefaction in small batch system (tubing bomb[4]).
7. Combustibility by burning in a thermogravimetric analyzer (combustion profile technique[5]).
8. Total oxygen by instrumental neutron activation analysis (INAA).

A number of internal checks described below were applied to the standard coal library data to ensure that all basic compositional analyses were accurate and meaningful.

Duplicate Analysis Scrutiny

All standard ASTM tests of elementary composition and for proximate analyses were done in duplicate. Samples were reanalyzed if the differences between duplicates exceeded ±2 standard deviations of the mean difference of all coals. If the rerun was still outside ±2 standard deviations, the test was repeated until the error was corrected. About 10-20% of the standard data required reanalysis, a figure which we believe would be typical for any well-run coal analysis laboratory. The data which were used for subsequent manipulation in the library were the arithmetic averages of two "best" duplicate runs.

Determined Versus Calculated Calorific Value

The calorific value of a coal can be calculated from the elementary composition and this value can be compared to the experimentally determined value as a check on the accuracy of both elemental and calorific value analyses. We checked the coal library data using a combination of three different formulae, two obtained from the literature and one derived specifically from the coal library samples.

*All on % dry coal basis except for moisture and calorific value which is Btu/lb on dry coal.

The Mott-Spooner formula calculates a dry, mineral matter-free (dmmf) calorific value (Btu/lb) using elemental analyses on a dmmf basis as:

$$CV_{MS} = 144.54 * C_{dmmf} + 610.2 * H_{dmmf} - 62.46 * O_{dmmf} + 40.5 * S_{org_{dmmf}} \quad (1)$$

If the dmmf oxygen content is greater than 11%, the Mott-Spooner Btu calculation (CV_{MS}) is modified by:

$$CV_{MS}\ (O_{dmmf} > 11\%) = CV_{MS} + [(0.31 * O_{dmmf} - 3.42) * O_{dmmf}] \quad (2)$$

Another formula was derived by the Institute of Gas Technology in Chicago as follows[6]:

$$CV_{IGT} = 146.58 * C + 568.78 * H + 29.4 * S - 6.58 * A - 51.53\ (O + N) \quad (3)$$

where C, H, N and S represent total dry carbon, hydrogen, nitrogen and sulfur respectively (dry basis), and A is the standard ASTM ash yield (dry basis). O+N is obtained by difference (100 - all other factors). The IGT formula gives the calorific value of the whole, dry coal whereas the Mott-Spooner gives the calorific value of the organic matter only. We derived our own formula using a stepwise regression for the 66 coals in the library. The basis for this formula differs somewhat from that used in the IGT or the Mott-Spooner formulae in that it yields a calorific value (Btu/lb) for dry coal from "corrected" dry analyses (an explanation of the corrections is discussed in a later section).

$$CV_{ERE} = 151.31 * C_{org} - 47.87 * O_{org} + 549.74 * H_{org} + 68.96 * S_{pyr} + 47.58 * S_{org} - 400.24 \quad (4)$$

Using the above equations we calculated the calorific values for all 66 coals and compared them to the determined calorific values. If the differences exceeded ±250 Btu/lb, the analyses were evaluated for errors and where appropriate, the samples reanalyzed. Table 3 summarizes the differences between determined and calculated calorific values using the Mott Spooner, IGT, and the Exxon Research (ER&E) formulae.

Table 3

COMPARISONS OF DETERMINED VERSUS CALCULATED CALORIFIC VALUE/(BTU/lb)

Formula	Mean Diff.	Std. Dev.	Value Range Min.	Max.
Mott Spooner (dmmf)	-59.3	113.6	-43.	213
IGT (dry)	-9.2	110.9	-399.	167
ER&E (dry organic)	-7.8	95.4	-298.	205.

All but a few of the coals gave excellent comparisons of calculated versus determined calorific values. We have not been able to determine why the few coals appear as outliers, even after re-analysis.

Elemental Balances

Total oxygen on dry coal can be determined independently using instrumental neutron activation analysis (O_{inaa}). Total oxygen can also be calculated by difference from dry analyses as:

$$O_{diff.} = 100\text{-}C\text{-}N\text{-}H\text{-}S_t\text{-}Cl\text{-}Ash\ Elements \quad (5)$$

where ash elements are the sum of the Si, Al, Fe, Mg, Ca, K, P, Na, and Ti calculated as a percentage of dry coal, and S_t is total sulfur. A comparison of the oxygen by difference against the oxygen by neutron activation serves as an independent check on the accuracy of the combined elemental analyses. The overall mean difference for the 66 coals was -1% with a standard deviation of 0.93 about the mean. The slight negative bias is probably due to the absence of minor and trace elements in the material balances. Several of the samples show significant deviations even though the elementary analysis appears to be valid. We believe the discrepancy may be due to moisture fluctuations or to interferences in the neutron activation analysis of oxygen.

Ash Checks

Ash determinations were done in duplicate on both the 16x100 mesh and the -60 mesh fractions. For all 66 coals, the minimum and maximum differences were -0.43 and +0.30 and the overall mean difference in ash between the two fractions was -0.089. The standard deviation was 0.15, well within the ASTM repeatability limit of 0.3.

CALCULATION OF DATA TO VARIOUS BASES

As-analyzed data are seldom of any direct use. Most raw analytical data must be calculated to some more meaningful basis in order to be effectively employed. All of our data were calculated to the dry basis. Because in most instances we were interested in properties and responses of the organic fraction, data were also calculated to a dry, mineral-matter free basis as described below.

Determination of the Mineral Matter Content

We estimated the inorganic matter (so-called mineral matter) content for the library coals from adjustments to the high temperature ash yield. The formula should apply to coals of all ranks and to coals that contain a variety of inorganic materials. Mineral matter content is calculated using the following relationship:

$$MM = Ash + H_2O_{clay} - 2.5(S_{ash}-S_{SO_4}) + 0.626 * S_{pyr} + CO_2 - O_{ie} \quad (6)$$

In Equation (6) the high temperature ash yield (Ash) is corrected using terms for the water of hydration of clays (H_2O_{clay}), the net amount of sulfate fixed in the ash ($S_{ash} - S_{SO_4}$) expressed on the coal basis, the decomposition of pyrite ($0.626 \times S_{pyr}$), the decomposition of carbonates to oxides (CO_2 loss) and a correction for the amount of organic oxygen that is retained in the ash (O_{ie}) owing to partial decomposition of humate salts in the lower rank coals. Other reactions of inorganic species during ashing are assumed to be negligible.

All but the H_2O_{clay} and O_{ie} are determined directly. S_{SO_4} refers to sulfate sulfur in the coal; CO_2 is determined according to ASTM D1756. The water of decomposition of clays is estimated using these relationships:

$$H_2O_{clay} = 0.10 * CLAY \quad (7)$$

$$CLAY = ASH - 1.2452 * S_{pyr} - 1.274 * CO_2 - 1.280 * Fe_{acid\ sol.} - 2.5 * S_{ash} - QTZ - ALK \quad (8)$$

Expression (8) approximates a clay content (CLAY) by subtracting estimates for the contributions of pyrite, carbonates, iron and sulfur oxides, quartz and any organically derived alkali oxides in the ash. Ten percent of the clay (Eq.7) is

then assumed to be the average water of decomposition. The organically derived alkali oxides (ALK) for use in Equation (8) are estimated from the analyses of the acid soluble alkalies by:

$$ALK = (CaO_{net} + MgO + Na_2O + K_2O)_{acid\ sol.} \quad (9)$$

where CaO_{net} represents all of the acid soluble Ca that is not stoichiometric with the amount of carbonate, estimated from the CO_2 yield as:

$$CaO_{net} = CaO_{acid\ sol.} - 1.274 * CO_2 \quad (10)$$

The ash element analysis can be used to estimate excessive amounts of quartz as:

$$QTZ = 2.1393 * [Si - (2.089 * Al)] \quad (11)$$

Equation (11) assumes that all aluminum is clay-associated and compensates only for coals that contain an exceptionally high content of free SiO_2.

A large portion of the alkali metals in lower rank coals are exchanged to oxygen functional groups. When coal is ashed, at either low or high temperature conditions, these organic/inorganic complexes decompose to yield alkali salts. Therefore, some of the oxygen in the ash is actually derived from organic oxygen. This can be approximated by summing the oxygen that would be stoichiometrically associated with the exchangeable alkali metals present on the coal. These are estimated from acid soluble data by:

$$O_{ie} = [O_{CaO_{net}} + O_{MgO} + O_{NaO} + O_{K_2O}]_{acid\ sol.} \quad (12)$$

The O_{ie} term in the mineral matter Equation (12) should be valid for the total range of coals. Most of the alkali metals in lignites, that are acid soluble, are bound to the organic matter. For the high rank coals, we found that by compensating for carbonate the O_{ie} term in the mineral matter expression is negligible.

Low Temperature Ashing

Mineral matter contents were also determined directly for the coals in the library using low temperature plasma oxidation[7], a technique which produces an inorganic residue from coal in more or less unaltered state. Most of the major minerals do not decompose under carefully controlled LTA conditions. Figure 3 shows a comparison between the calculated and experimental (LTA) mineral matter contents of the coals. In the figure, data points with an "x" refer to low rank coals whose oxygen contents are greater than 16% (dmmf). When these coals are excluded, there is good agreement between the two methods; the mean difference for the 52 higher rank coals is essentially zero (-0.01), but with fair amount of scatter (the standard deviation is 0.85).

As shown in Figure 3, the LTA yield of the high oxygen coals is much greater than the calculated mineral matter content. Lignites and subbituminous coals contain appreciable alkali cations bound to oxygenated functional groups. These tend to inhibit the oxidation process, preventing complete ashing; thus, the LTA yields tend be too high. Also, a substantial amount of combustion gas (as either S- or N-oxides) adds to the weight of the LTA by reacting with the molecularly dispersed alkalies. When these coals are extracted with acid, the alkali cations are removed, and no analytical difficulties are encountered with the LTA

technique. Thus, the best direct measure of the mineral matter content in low-rank coals is the sum of the yield of LTA on acid-extracted coal and the weight fraction of acid-soluble inorganics, or

$$MM_{acid+LTA} = CLTA^* (100-A/100) + A \quad (13)$$

where CLTA is the LTA yield on dry, acid-washed coal and A is the weight loss upon acid extraction.

Figure 4 is a plot of the mineral matter calculated from Equation (6) against the mineral matter from the modified LTA method (13). Note that significant improvement is seen with the high-oxygen coals (indicated by "X"). The summary statistics of mean differences indicates that the modified LTA method for estimating the mineral matter content is unaffected by differences in rank.

The formula-derived mineral matter content is applicable to all ranks; to maintain consistency within the library, we used the formula for all calculations to the dmmf basis.

Volatile Matter and Calorific Value

The ASTM volatile matter yield, determined at 950°C, includes components from decomposition of inorganic materials. To obtain an organic volatile content we used a formula modified from Leighton and Tomlinson[8] where

$$VM_{corrected} = VM_{dry} - H_2O_{clay} - 0.41 * S_{pyr} - 0.9 * CO_2 - 0.76 * Cl \quad (14)$$

This formula compensates for volatile loss of clay water, pyritic sulfur, carbonate CO_2, and chlorine.

Calorific value (Btu/lb) determinations should be corrected for contributions due to the exothermicity of pyrite oxidation, thus,

$$CV_{corrected} = CV_{dry} - 55.67 * S_{pyr} \quad (15)$$

Fixed carbon content on the dmmf basis is calculated as:

$$FC_{dmmf} = 100 - VM_{dmmf} \quad (16)$$

Elemental Analyses

Carbon and hydrogen determinations by the ASTM method include carbonate carbon and clay water hydrogen respectively. Corrections to obtain the organic carbon and hydrogen contents are made as:

$$C_{org} = C_{dry} - 0.2729 * CO_2 \quad (17)$$

$$H_{org} = H_{dry} - H_{clay} \quad (18)$$

Where $H_{clay} = 0.1119 * H_2O_{clay}$ estimated from equations (6) and (7).

$$O_{dmmf} = 100 - H_{dmmf} - C_{dmmf} - N_{dmmf} - Cl_{dmmf} - S_{org_{dmmf}} \quad (19)$$

Corrected analyses are multiplied by the factor 100/(100-MM) to convert to the dmmf basis.

To express the calculated organic oxygen on the dry basis (O_{org}) the O_{dmmf} value is multiplied by (100-MM)/100.

CORRELATIONS

The purpose of the coal characterization research program is to establish relationships between fundamental coal properties, derived coal properties, and process responses. Many novel relationships have been found employing multivariate statistical analysis techniques. These results will be reported in publications to follow. Three examples of the kinds of correlations that have been developed are reported below.

Coal density by helium pycnometry is related to the elemental composition as defined by Equation (22).

$$D_{He(dmmf)} = 0.023 * C_{dmmf} + 0.0292 * O_{dmmf} - 0.0261 * H_{dmmf} + 0.0225 * S_{org_{dmmf}} - 0.765 \quad (22)$$

The above expression accounts for 94% of the variance of the densities of the samples (viz. $r^2 = 0.943$).

For those coals exhibiting a free swelling index (FSI) greater than zero, 91% of the variance of the FSI for the samples can be explained according to Equation (23).

$$FSI = 0.875 * C_{dmmf} + 0.859 * S_{dmmf} + 1.304 * H_{dmmf} + 0.347 * R/I - 77.715 \quad (23)$$

In Equation (23), R/I refers to the ratio of reactive macerals (vitrinite, liptinite) to "inert" macerals (fusinite, micrinite) plus mineral matter.

Volatile matter is also strongly correlated with elemental composition ($r^2 = 0.96$) according to the relationship shown in Equation (24).

$$VM_{dmmf} = 1.281 * O_{dmmf} + 12.345 * H_{dmmf} + 1.915 * S_{org_{dmmf}} - 42.251 \quad (24)$$

CONCLUSIONS

Procedures for estimating responses of coals in synthetic fuels conversion processes are practically nonexistent. Consequently, a new look at coal characterization and classification procedures is propitious. We believe that meaningful understanding of the relationships between coal properties can be obtained only through the study of a large suite of carefully selected, prepared and analyzed coal samples. We have, therefore, begun a comprehensive coal characterization research program at the Baytown, Texas, laboratory of Exxon Research and Engineering Company. Sixty-six fresh samples of coal representing the coalification band in an H/C vs O/C plot have been analyzed in detail. Eighty percent of the samples contain <10% mineral matter. Eighty percent also contain >80% vitrinite. Through meticulous preparation procedures designed to minimize exposure to air, through numerous cross-checks of validity of the analytical data, and through calculation of the data to a mineral-matter-free basis we have assembled a data library that is being used to define interrelationships between coal properties. Numerous multivariate correlations have been found indicating strong dependence of properties such as density, free swelling index and volatile matter on elemental composition. Such correlations promise to shed considerable light on the area of coal characterization and classification.

References

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FIGURE 1

DISTRIBUTION OF BAYTOWN COAL CHARACTERIZATION LIBRARY SAMPLES ON VAN KREVELEN PLOT

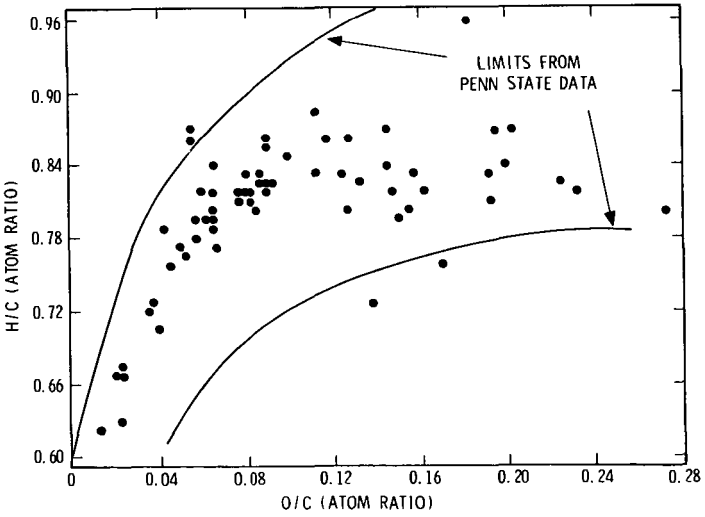

FIGURE 2. BAYTOWN COAL LIBRARY PREPARATION

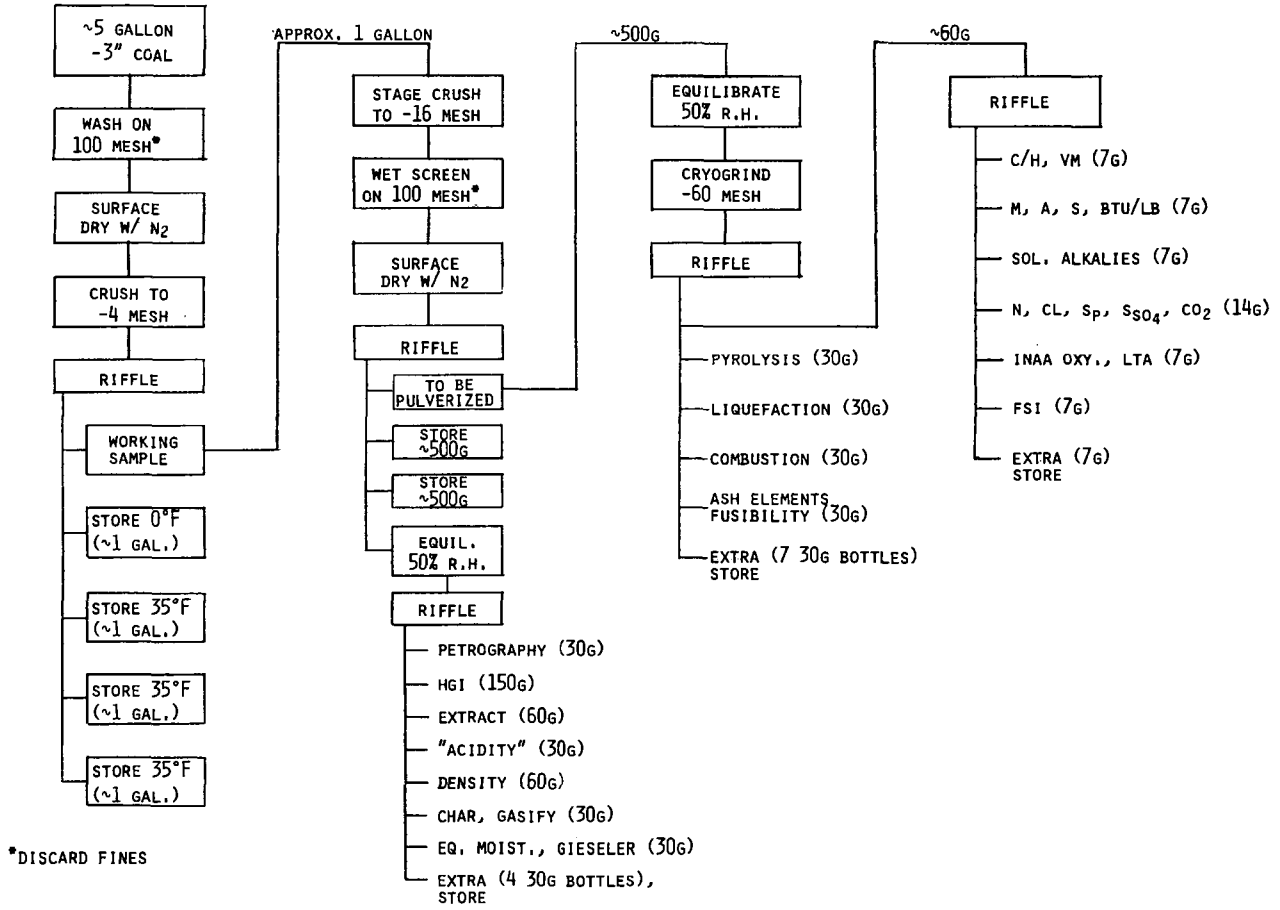

FIGURE 3

RELATION BETWEEN THE LOW TEMPERATURE ASH YIELD AND THE CALCULATED MINERAL MATTER CONTENT

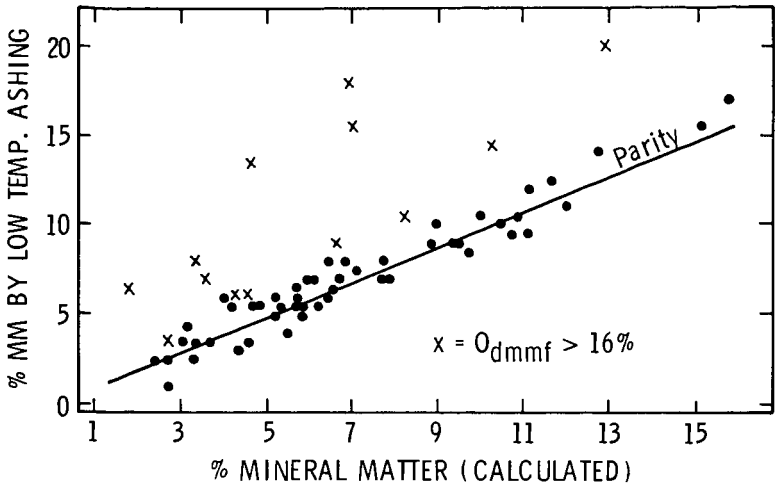

FIGURE 4

RELATION BETWEEN THE MINERAL MATTER CONTENT BY THE ACID MODIFIED LTA METHOD AND THE CALCULATED MINERAL MATTER CONTENT